



CASE CO/21-22865/A/PCT

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4/30/09
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE PCT NATIONAL STAGE APPLICATION OF
PETER NESVADBA ET AL.

Group Art Unit: 1625

Examiner: OH, TAYLOR V

INTERNATIONAL APPLICATION NO. PCT/EP
2004/050315

FILED: MARCH 17, 2004

FOR: PROCESS FOR THE OXIDATION OF
SECONDARY AMINES INTO THE
CORRESPONDING NITROXIDES

U.S. APPLICATION NO: 10/549,526

35 USC 371 DATE: SEPTEMBER 19, 2005

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

APPEAL BRIEF

Sir:

This appeal is from the final rejection of claims 1-10 mailed from the PTO on October 30, 2008. A Notice of Appeal was mailed on January 30, 2009. This Appeal Brief is filed along with a request for a 1 month extension of time.

The Commissioner is authorized to charge any fee due, or credit any overcharge, as a result of this Amendment to Deposit Account No. 03-1935.

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(1) REAL PARTY OF INTEREST

The real party of interest, by virtue of an assignment recorded in the United States Patent and Trademark Office on July 31, 2006 reel/frame 018139/0051 is:

Ciba Corporation (formally Ciba Specialty Chemicals)
P.O. Box 2005
540 White Plains Road
Tarrytown, New York 10591

(2) RELATED APPEALS AND INTERFERENCES

Appellant is not aware of any related appeals and interferences for the above application.

(3) STATUS OF THE CLAIMS

Claims 1-10 are pending. Claims 1-10 are rejected and are presented for appeal.

(4) STATUS OF AMENDMENTS

The amendments presented After Final mailed on January 30, 2009, were not entered by the examiner. Thus the status of the claims are as amended on July 17, 2008.

This brings up to date the status of the claims. A clean copy of the claims along with status identifiers is found in an attached appendix.

(5) SUMMARY OF THE CLAIMED SUBJECT MATTER

Claim 1 is the only independent claim. Claims 2-10 depend from claim 1.

Claim 1

Claim 1 is directed to:

A process for the preparation of secondary nitroxide radicals from their corresponding secondary amines by oxidation with an organic peracid, comprising the steps

- a) adding to a reaction vessel a secondary amine, optionally together with an organic solvent, and in one batch a base selected from the group consisting of alkali metal bicarbonates, alkaline earth metal bicarbonates, ammonium bicarbonates, alkaline earth metal carbonates ammonium carbonates and mixtures thereof in the form of a solid together with water or as an aqueous slurry;
- b) dosing a peracid under stirring to the reaction mixture in an amount of 1.0 to 2.5 mol per mol of secondary amine; and
- c) isolating the organic phase.

Support for the above independent claim may be found in the Specification on page 2, lines 14-23. Adding "a base in one batch" is defined in the Specification on page 2, lines 24-25 to mean that the total amount is added in one single batch, in contrast to dosing it in small portions.

Support for the added elements of the dependent claims is found in the specification as follows:

Claim 2, wherein the organic solvent optionally used in step a) is immiscible with water, page 2 line 26;

Claim 3, wherein the peracid is peracetic acid, page 3 line 2;

Claim 4, wherein the amount of water added in step a) is sufficient to dissolve the organic acid salt formed in the neutralization reaction between the organic acid and the bicarbonate or carbonate, page 3 lines 5-8;

Claim 5, wherein the base is sodium or potassium bicarbonate, calcium or magnesium carbonate or dolomite, page 5 lines 3 and 4 following the table;

Claim 6, wherein the base is added in an amount of from 0.1 to 1.5 equivalents base per 1 equivalent of all acids present, page 5 lines 6-7 following the table;

Claim 7, wherein the reaction temperature is between 0° C and 40° C, page 5 lines 8-9 following the table

Claim 8, wherein the dosage of the peracid is carried out from 10 minutes to 5 hours, page 5 line 10;

Claim 9, the formula at the top of page 6;

Claim 10, formula and description bottom page 6 through the of top page 7.

(6) GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

1. Claims 1-10 stand rejected under 35 USC 103(a) as obvious over Gillet et. al., US 6,538,141.
2. Claim 10 is rejected under 35 USC 112 second paragraph for using improper open language.

(7) ARGUMENTS

Claim 1-10 Arguments Regarding Obviousness

1. Claims 1-10 stand rejected under 35 USC 103(a) as obvious over Gillet et. al., US 6,538,141.

US 6,538,141 discloses a process wherein a solution of a peracid and a separate solution of a base, for example an aqueous solution of an alkali metal carbonate or bicarbonate, are simultaneously added to a two phase emulsion of water and amine which is pre-dissolved in an appropriate organic solvent. The solutions of peracid and base are added in portions and are regulated in such a way that the pH is maintained in the range of 4 to 12, column 3, lines 4-14 of '141.

In the instant process, the base is added in total as a solid or slurry to the amine and an optional organic solvent at the beginning of the reaction. The base is not dissolved in water, thus limiting the amount of water needed. The peracid is then added to this mixture.

Appellants respectfully maintain that there is a significant difference in the two processes in that the process of '141 adds a solution of base at intervals during the reaction with peracid and the instant process adds all of the base at once in the form of a solid or as a slurry. Appellants claim that the instant process affords the advantages a simpler and effective means of pH control and of vastly reducing the water needed during the reaction, thus greatly reducing the volume of the reaction and significantly lowering costs of production.

The Examiner notes that both processes use a peracid to oxidize an amine and uses a base to control the pH. The examiner also holds that '141 can be read so as to suggest the use of Appellants' "solid base in the presence of water or slurry" given the generic teaching which describes an "aqueous solution of a carbonate or hydrogen carbonate", bottom paragraph of page 4 of the Advisory Action of February 25, 2009. The top paragraph of page 4 of the Action notes that '141 realizes the desirability of limiting the volume of water as "efforts will be made to use that are as concentrated as possible". On page 5 of the Action the Examiner states that when using a slightly soluble base in the '141

process, such bases can partially remain in solid or slurry form and that therefore. The artisan would be motivated to use a base in solid or slurry form to control the pH.

Regarding whether Gillet, US 6,538,141 discloses the potential use of a solid base, Applicants respectfully point out that not only is the prior art silent regarding the use of a solid base, the process of the prior art excludes the use of a solid base for at least the following reasons. In the prior art process, the base is added as a solution in portions simultaneously with the addition of the peracid to maintain a particular pH profile during the course of the reaction. Significantly, Gillet is clear that the base is added as a solution, column 3, lines 22-27, "The weight concentration of the aqueous solutions of alkali metal or alkaline-earth metal carbonates or hydrogen carbonates is set by the solubility limit of these species in water" and even though "Efforts will be made to use solutions that are as concentrated as possible", only solutions are to be used and the amount of water present in the invention will necessarily be at least as much as is needed to dissolve the entirety of the base. Thus, while the art recognizes the need to reduce water, there is no suggestion how to accomplish this other than using a highly concentrated solution.

Regarding whether it would be obvious to use a solid base or slurry in view of the teaching of the cited art, Appellants note the significance of pH control as discussed in the instant Specification on page 1 lines 19-24. Appellants further respectfully point to page 2 of the instant Specification, lines 3-13 where some of the difficulties of the process of the cited art are listed, in particular, reference is made to the difficulty of accurately determining pH in a heterogeneous mixture, such as the emulsion of '141.

Thus, the presence of the correct amount of base available to the reaction is an essential component to the successful and efficient conversion to nitroxyl. Any process must therefore comprise a way of controlling the pH. In '141, an amount of base in solution is added along with the peracid. Care is taken to make sure that the correct amount of base is added.

When using a base in solution added to an emulsion, one can assume that with good mixing an appropriate amount of base will be available in a usable form to efficiently neutralize the acid that is formed. Appellants respectfully assert that until the instant invention, there is no guarantee that the solid, undissolved base will be reactive enough in that form to adequately control the reaction. Appellants have found, quite surprisingly, that having the base present as a solid or slurry and not as part of a solution effectively controls the amount of base released to the reaction. Significantly, no

special care needs to be taken to make sure that the right amount of base in solution is added at the proper moment as in '141.

Appellants' therefore submit that the use of base in solid or slurry form is not an obvious choice in light of the cited art and that the ease of pH control afforded by the instant process is a significant and unexpected result.

To elaborate further on the issue of water volume, Appellants respectfully aver that this is not an insignificant issue and refer to page 4 and 5 of the instant Specification and the instant examples. Table 2 on page 5 shows that the amount of water required can be reduced by 80% or more, providing large savings in manufacturing by increasing greatly the volume yield. Further, the instant process allows one to use some bases that can not be used in a practical manner following the method of '141 because they have such low solubility in water that solutions with practical concentrations can not be prepared, for example CaCO_3 , MgCO_3 or Dolomite ($\text{CaCO}_3 \times \text{MgCO}_3$).

Appellants therefore respectfully submit that the process of instant claim 1 differs significantly from that of Gillet, US 6,538,141 in the use of base in the form of a solid or slurry. Appellants respectfully maintain that Gillet, US 6,538,141 requires the use of a base in solution which is attested to both by the manner in which the base is added and by the disclosure in column 3, lines 22-27 of Gillet that "The weight concentration of the aqueous solutions of alkali metal or alkaline-earth metal carbonates or hydrogen carbonates is set by the solubility limit of these species in water". Appellants further respectfully maintain that until demonstrated by the success of the instant invention one could not anticipate that the use of a solid base or slurry could adequately substitute for the solution of Gillet, US 6,538,141.

Appellants respectfully stress that there are significant differences in reactivity, rates, and processing requirements that can be encountered between using a base that is a solid and a base in solution.

Appellants further respectfully submit that the invention of instant claim 1, and all dependent claims, unexpectedly provides a significantly more effective and more easily controlled process for proper pH control of the instant reaction. Further benefits such as simpler reaction vessel design, less complicated processing steps and the higher volume yield enabled by eliminating much of the water required in the '141 process are additional advantages afforded the industry by the invention.

In light of the above discussions, Appellants respectfully submit that the rejections of claims 1-10 under 35 USC 103(a) over Gillet et. al., US 6,538,141 have been rebutted and respectfully ask that the rejections be reconsidered and reversed.

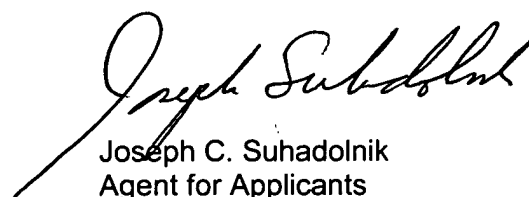
Claim 10 Arguments Regarding Indefiniteness

2. Claim 10 stands rejected under 35 USC 112 second paragraph for using improper open language.

The examiner rejects the claim as the word "the nitroxide radical contains" suggests that there are additional components besides the nitroxyl. Appellants respectfully point out that the "nitroxide radical" is clearly a part of a compound, which compound necessarily contains other structural elements. Claim 10 merely limits the compound prepared by the process of claim 1 to those compounds in which the N-O functionality is contained within a structural element of formula X. Appellants maintain that open language is proper and required in that there are other components which make up the compound being prepared.

Appellants respectfully submit that the rejections of claim 10 under 35 USC 112 second paragraph for using improper open language have been rebutted and respectfully ask that the rejections be reconsidered and reversed.

Respectfully submitted,



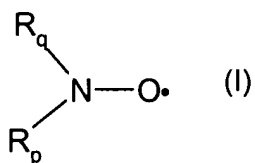
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Enclosure Transmittal Letter, Claims Appendix (8), Evidence Appendix (9),
Proceedings Appendix (10).

(8) CLAIMS APPENDIX

1. (previously presented): A process for the preparation of secondary nitroxide radicals from their corresponding secondary amines by oxidation with an organic peracid, comprising the steps
 - a) adding to a reaction vessel a secondary amine, optionally together with an organic solvent, and in one batch a base selected from the group consisting of alkali metal bicarbonates, alkaline earth metal bicarbonates, ammonium bicarbonates, alkaline earth metal carbonates ammonium carbonates and mixtures thereof in the form of a solid together with water or as an aqueous slurry;
 - b) dosing a peracid under stirring to the reaction mixture in an amount of 1.0 to 2.5 mol per mol of secondary amine; and
 - c) isolating the organic phase.
2. (original) A process according to claim 1 wherein the organic solvent optionally used in step a) is immiscible with water.
3. (original) A process according to claim 1 wherein the peracid is peracetic acid.
4. (original) A process according to claim 1 wherein the amount of water added in step a) is sufficient to dissolve the organic acid salt formed in the neutralization reaction between the organic acid and the bicarbonate or carbonate.
5. (original) A process according to claim 1 wherein the base is sodium or potassium bicarbonate, calcium or magnesium carbonate or dolomite.
6. (original) A process according to claim 1 wherein the base is added in an amount of from 0.1 to 1.5 equivalents base per 1 equivalent of all acids present.
7. (original) A process according to claim 1 wherein the reaction temperature is between 0° C and 40° C.
8. (original) A process according to claim 1 wherein the dosage of the peracid is carried out from 10 minutes to 5 hours.
9. (original) A process according to claim 1 wherein the nitroxide radical is of formula (I)

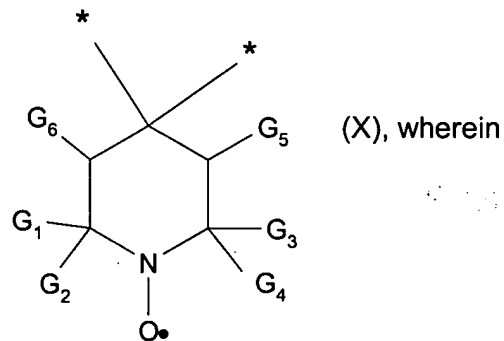


R_p and R_q are independently tertiary bound C_4 - C_{28} alkyl groups or C_3 - C_{17} secondary bound alkyl groups which are unsubstituted or substituted by one or more electron withdrawing groups or by phenyl; or

R_p and R_q together form a 5, 6 or 7 membered heterocyclic ring which is substituted at least by 4 C_1 - C_4 alkyl groups and which may be interrupted by a further nitrogen or oxygen atom.

10. (previously presented) A process according to claim 1 wherein the nitroxide radical is a

compound which contains a structural element of formula (X)



G_1 , G_2 , G_3 , G_4 are independently C_1 - C_6 alkyl or G_1 and G_2 or G_3 and G_4 , or G_1 and G_2 and G_3 and G_4 together form a C_5 - C_{12} cycloalkyl group;

*denotes a valence; and

G_5 , G_6 independently are H or C_1 - C_6 alkyl.

(9) EVIDENCE APPENDIX

Not Applicable.

(10) RELATED PROCEEDINGS APPENDIX

As the appellants are not aware of any other related proceedings, no copies of decisions rendered by a court or the board are attached.